SUMMARY OF THE
QUALITY SYSTEMS COMMITTEE MEETING
ANNAPOLIS, MD
NOVEMBER 8-10, 1998

The Quality Systems (QS) Committee met November 8, 9, and 10, 1998, in Annapolis MD. The meeting was led by its chair, Mr. Joe Slayton. Each day's session is summarized below. A list of action items from all three days is given in Attachment A. A list of participants from all three days is given in Attachment B. A parking lot for issues to be addressed at a later date is given in Attachment C. The QS Committee's method for handling written comments is given in Attachment D. A list of comments to be addressed is given in Attachment E. Comments assigned to QS Committee participants and their responses are given in Attachment F.

SESSION 1
November 8, 1998

The QS Committee met on Sunday, November 8, 1998, at 10 a.m. Eastern Standard Time (EST) at the EPA Laboratory facility in Annapolis, MD. *The purpose of this session was to discuss instrument calibration and detection and to address committee business and administrative items.*

ADMINISTRATIVE ISSUES

The QS Committee discussed how to manage and address the incoming comments. Appendix D presents the approach to handling the received comments along with a template for commentors to use when submitting comments to the committee.

The committee established a set of criteria by which to evaluate the requirements specified in Chapter 5. The standards in Chapter 5 should meet the criteria listed below.

Flexible: Allow laboratories freedom to use their experience and expertise in performing their work and allow for new and novel analytical methods and approaches, (e.g., Performance Based Measurement System [PBMS]). That the standards specify the "What" and avoid were possible the "How To", (e.g., control limits must be developed to determine if a quality control (QC) check result is acceptable, the standards do not specify how the laboratory is to determine these limits).

Auditable: Sufficient detail is included so that the accrediting authorities evaluate laboratories consistently and uniformly.

Practical: The standards represent essential quality assurance (QA) policies and QC procedures and that these standards should not place an unreasonable burden upon the laboratories.

Internationally Applicable: Consistent with ISO Guide 25

DISCUSSION OF SECTION 5.9.4, INSTRUMENT CALIBRATION

When completed with Section 5.9.4 the QS Committee will review Sections 5.9.1, 5.9.2, 5.9.3 and 5.8 for redundancy and determine whether any of these sections can be combined.

The specific discussion of what is fundamental to achieving sterilization in autoclaves will be moved from 5.9.4.1.2 to Appendix D.3 because theses requirements are specific to the needs of microbiological analysis. The specific wording will be addressed when Section D.3 is reviewed. Autoclaves were still included in the requirements for calibration of support equipment in Section 5.9.4.1.

Section 5.9.4.2.1.f was reworded to make it clear that data qualifiers or flags must have an explanation.

Section 5.9.4.2.1.g was reworded to make it clear that data associated with an unacceptable initial calibration curve cannot be reported.

Section 5.9.4.2.1.j, regarding maintaining instructions from equipment manufacturer for initial instrument calibration, was deleted because it is already addressed in Section 5.8.e.

Section 5.9.4.2.1.c was reworded to make it clear that quantitation of sample results must be done using the initial calibration curve and not a calibration verification check.

Section 5.9.4.2.2.e allows non-detection results associated with a high bias continuing calibration curve to be reported. The QS Committee felt that reporting should also be allowed for the opposite situation. That is, a situation where results that exceed a regulatory limit or decision level are associated with a low bias continuing calibration curve. In both situations, the data must be qualified. The QS Committee decided to allow reporting for the low bias condition because the consequence of a decision error for this situation is potentially less severe than for the high bias situation. That is, the data indicate an exceedance when the true level is below the established limit. This may be of less concern than not detecting a substance when it is actually present at a level that may be of concern.

Two requirements were added to Section 5.9.4.2.2. First a continuing calibration check must be repeated at the end of each analytical batch as well as at the beginning. The QS Committee felt that this should be a minimum requirement to determine that an instrument is still within the initial calibration range at the end of an analytical batch. Second, the calibration verification checks must include concentrations at the lowest and highest concentration of the initial instrument calibration. The option of substituting the lowest regulatory limit associated with the samples in the analytical batch in place of the lowest concentration from the initial calibration curve was included. The QS Committee felt that the initial calibration should be verified over its entire range.

The question was raised as to what constitutes an analytical batch for a continuous monitoring process. This may also be an issue for the Field Measurements and Sampling Committee to address. The Committee decided to revisit this issue at a later time. Addressing this issue in

Chapter 5 may require a separate section. A related issue was raised regarding calibration checks for continuous monitoring. The QS Committee felt that calibration checks could be periodically introduced to the monitoring system. The frequency of checks may depend on the stability of the analytical instrument.

DISCUSSION SECTION D.1.4 DETECTION LIMITS

In Section D.1.4.a. online analysis (not necessarily continuous monitoring) was added, to address air monitoring, as an example of a component for which an Method Detection Limit (MDL) study is not required because spiking solutions are not available.

The following points were raised during the QS Committee discussion of detection limits.

- Should 40 CFR Part 136, Appendix B be included as the protocol for determining the method detection limit (MDL) when the mandated test method or applicable regulations do not specify a protocol? This requirement may be too prescriptive and not provide sufficient flexibility. However, the existing language in Appendix B does allow for some flexibility depending on an instrument's capabilities. If a regulating agency wants a protocol besides Appendix B used, they can specify that protocol in their regulations.
- Using the protocol in Appendix B could be made a recommendation with a list of reasons for its use. Another approach would be to develop a list of essential elements to follow when determining an MDL. A list could be derived from the common elements in other standards such as Appendix B and ISO Guide 25. However, the issue of MDL determination is statistically complex and contentious. Trying to make a list of essential elements may miss some import elements and not make for a good standard.
- Some argue that determining an MDL is not necessary. For instance, in situations where one is not interested in detecting low levels of a substance, but is concerned with higher levels that exceed a specified limit. However, even in such cases, the MDL gives an idea of the lower range detection capabilities. Also, MDLs could be a factor in selecting a laboratory to perform the analysis. One option would be to allow high level MDLs if measuring at lower level is not a concern.
- The level obtained when determining an MDL may not be representative of what can be obtained during normal operations. For instance, the analytical instrument may be cleaned especially for the MDL study. In addition, the MDL can vary according to the equipment and the technique used.

SESSION 2
November 9, 1998

The QS Committee met on Monday, November 9, 1998, at 9:00 a.m. Eastern Standard Time (EST) at the US Fish and Wildlife facility in Annapolis MD. *The purpose of this session was to discuss instrument calibration and detection, obtain Environmental Monitoring Management Council (EMMC) and audience input on calibration and detection, and discuss comments received on Chapter 5.*

DISCUSSION OF SECTION 5.9.4, INSTRUMENT CALIBRATION

Section 5.9.4.1.a was revised to specify that records of repairs and maintenance must be kept as well as records of service calls. This change was intended to cover in-house maintenance as well as service calls by outside technicians.

Section 5.9.4.2.2.f was revised to specify that a second consecutive calibration check means one performed immediately following the failed calibration verification.

The comment was made that Section 5.9.4.2.2.f.ii should capture the idea that individual samples are independent of one another and not all the samples in a batch need to be at a high concentration to meet the condition of this section. In addition, the language was revised to include a "decision level" as well as a "regulatory limit." In addition, the parenthetical reference to high concentration was deleted as it isn't auditable and does not add meaning to the standard.

The comment was made, regarding Section 5.9.4.2.2.f.ii, that in cases where the data are averaged and compared to a regulatory level, allowing biased data may corrupt the average. However, the standard requires the data be flagged and some programs may not allow the use of flagged data when averaging. In addition, in some programs it is important to know how much the measured value exceeds the limit. The point was raised that data from a low bias curve may underestimate the actual level, which is a concern from a public health risk view.

The point was raised that there may not be an incentive to correct the low bias situation, which could result in higher measured values. Therefore, it may be advantageous to continue generating qualified data with a low bias.

Regarding Section 5.9.4.2.2.f.ii, the point was raised that even with a high bias continuing calibration curve, a sample measured as nondetect may actually be present at a detectable level, especially if the high bias is small.

COMMENTS FROM EMMC

Mr. Barnes Johnson (EPA, EMRAD), who is tri-chair of the EMMC Laboratory Accreditation Panel, presented the EMMC comments.

EMMC is a multilevel organization that tries to coordinate analytical methods and monitoring across the Agency. A memorandum was developed after the last NELAC meeting. All EPA offices were invited to participate in this process. The following six main issues were raised.

1. Method Detection and Calibration. Not all EPA programs require an minimum detection limit (MDL) to be established or find it of value since the regulatory limits are much higher than what is considered to be the MDL for the analytical methods. The concern is more for the sensitivity of the measurement around the value that is set for the regulatory limit.

2. Performance Based Measurement System. Chapter 5 Appendix E needs to be completed to address PBMS. It's not clear why there's a difference between Appendix C, Initial Demonstration of Capability and Appendix E, PBMS. These appendices could be combined.

3. Program Method Analyte. The change to program, matrix, analyte, is positive. Need to get away from the emphasis on method and more on matrix in light of PBMS.

4. Solid and Waste Matrices. There are unique procedures for handling these matrices that need to be dealt with. Appendix D, Essential Quality Control Requirements, seems to focus on water. The comment was made that the intent of the Committee was to draw upon common QA/QC procedures among the different programs to develop minimum criteria or essential items. The intent was not for Appendix D to reflect water issues. The Committee hasn't addressed cleanup or preparation steps for solid or waste matrices because they are dealt with in the method in SW 846. The intent of SW-846 was to provide a basic framework allowing flexibility, which should be reflected in Appendix D, Essential Quality Control Requirements.

5. Matrix Spikes. Matrix spikes may not be necessary in some cases. Bias still needs to be evaluated; however, matrix spikes may not be possible or useful in some cases.

6. Field Sampling and Measurement. Conceptually, field measurements should work the same as in the laboratory. However, certain things in field sampling and measurement are very difficult to do practically.

DISCUSSION SECTION D.1.4 DETECTION LIMITS

The comment was made that the concern is not the MDL but to demonstrate "sensitivity" at the regulatory level or level of concern. This could be considered a sensitivity check.

The QS Committee felt that it would be too difficult to develop a minimum standard procedure for determining the MDL because of the complex statistical issues involved. 40 CFR Part 136, Appendix B was originally include in this section because it was commonly used.

The comment was made that allowing the option to use any available procedures for determining the MDL would be to hard to audit because it would require the auditors to understand all the different procedures.

Other approaches proposed for addressing the MDL procedure are listed below:

- Use Appendix B but allow the spiking level and matrix to be appropriate to the intended application.
- Separate the discussion on MDLs between those programs that require it and those that do not.
- If an MDL determination is not required under a regulation then leave the procedure open as is done in ISO 17025. However, it may be difficult to audit such an open requirement and would require the auditors to be more sophisticated in evaluating the method used by the laboratory. However, the auditors role is to evaluate whether the laboratories do what they say they are doing and not to evaluate the MDL procedure. In addition, leaving the procedure open may negatively impact reciprocity because State programs may specify different procedures. However, leaving the procedure open may fit better with PBMS.

General language was developed for the opening paragraph of Section D.1.4 along with a list of six specific requirements (items a-f).

- Item b was modified to specify what a "clean" matrix is.
- Item c was revised to require that the quantitation limit for the test method must be established.
- Regarding item d, the question was raised as to what constitutes a significant change in the test method that would require the detection limit to be determined. The requirement could be that an MDL must be determined when there is a significant change in the procedure as for the initial demonstration of capability. However, as long as continuing calibration checks are performed at a low concentration level with acceptable results then an MDL determination may not be required. The requirement should include changes in instrument type. There can be significant differences within instrument type (e.g., types of columns on a GC) that may require a new MDL determination. The language in item d was made consistent with IDOC.
- The QS Committee needs to revisit the definition of quantitation limit after the MDL definition has been changed. The reference to 3.18 x MDL as a quantitation limit will be removed from the Chapter. The requirements should not specify a numerical value for the quantitation limit because it will depend on the preparation steps and matrix.
- Item e was revised to specify that procedures, data, and matrix type must be documented.

DISCUSSION OF WRITTEN COMMENTS

The following discussion was on written comments about Chapter 5 that the QS Committee received. The comments were divided among the Committee participants for initial review. Those comments that required additional discussion or a change to the Chapter were discussed by the entire QS. Refer to Attachment F for a listing of the numbered comments and each QS Committee participant's detailed responses to each comment. The action the QS Committee took on the comments is summarized below.

Comments C1 and C13
No changes were made to the Chapter

Comments C5 and C15
Regarding the comment to Section D.1.7.d, the QS Committee felt that the listed items are not measures of selectivity and other documentation requirements address this issue. No change was made to the Chapter.

Comment C6
No changes were made to the Chapter.

Comment C7
The QS's discussion focused on how a one person or a small laboratory will perform internal audits. The QS Committee decided to replace the language in Section 5.5.3.1 with language from ISO 17025 Section 4.13.1.

Comment C9
The QS Committee decided at the November 8, 1998 meeting that the subject of continuing calibration verification for continuous monitoring will addressed at a later date. This comment was added to the issues parking lot.

Comment C10
The QS Committee removed "temperature" from the list of examples in Section 5.11.3.a.2

SESSION 3
November 10, 1998

The QS Committee met on Tuesday, November 10, 1998, at 8 a.m. Eastern Standard Time (EST) at the US Fish and Wildlife facility in Annapolis MD. *The purpose of this session was to discuss written comments on Chapter 5 and the proposed changes from the Air Monitoring subcommittee.*

DISCUSSION OF WRITTEN COMMENTS

The following discussion was on written comments about Chapter 5 that the QS Committee received. The comments were divided among the QS Committee participants for initial review. Those comments that required additional discussion or a change to the Chapter were discussed by the entire QS. Refer to Attachment F for a listing of the numbered comments and each QS Committee participant's detailed responses to each comment. The action the QS Committee took on the comments is summarized below

Comments on Section D.1.1.b.4
The QS Committee felt that a 2 year period was a reasonable time for spiking all the components of a test method.

In addition the QS Committee discussed the allowance for spiking a representative number of components (10%) for test methods with an extremely long list of components. The points of discussion are listed below.

- The 10% allowance should be eliminated unless there is an interference problem with spiking all the components.
- Does the 10% allowance conflict with the requirement that the spiked components should represent all chemistries, elution patterns and masses? Also, what if a client requests analyses for more than 10% of the components of a test method?
- Requiring laboratories to spike for all the components may be an excessive burden, it may not be essential for quality control, and it may not be practical.
- Some laboratories may only perform analyses for the same set of components and it may not be necessary for them to spike for all the components.
- Does reportable mean that spikes are needed only for the components that are routinely analyzed for clients or does it mean any component that can be measured and reported.

Comment C11
No change was made to Chapter 5.

Comments C1, C12, C15, C16, and C17
No changes made to Chapter 5.

Comments C4 and C14
The QS Committee did not agree with comment on Section 5.9.4.3.d.

The QS Committee revised Section 5.9.4.4.2.c to address continuing and initial calibration verifications.

The QS Committee felt the terms in 5.12.4 are defined sufficiently.

The QS Committee did not agree with the comment on Section 5.9.4.2.1.c

The QS Committee felt that the suggested wording for Section D.1.1.1.a is more stringent and less flexible than the current language and that method blank subtraction is acceptable as long as it is required in method or regulations.

Regarding the comment on D.1.4.d, the QS Committee has already revised this section.

Comment C8
In Section 5.5.4.b, the QS committee replaced the term usability with validity and in Section 5.12 replaced appropriate period with a minimum of five years.

The point of the comment regarding the checklist was unclear as Chapter 5 does not have a checklist.

Section 5.4.2.j refers proficiency testing issues to Chapter 2.

DISCUSSION OF PROPOSED CHANGES FROM AIR MONITORING SUBCOMMITTEE

The charge to the QS Air Subcommittee, which is led by Mr. Glowacki, was to review Chapter 5 through Appendix D and look for changes that need to be made to accommodate air monitoring as well as editorial changes. The subcommittee's approach was to incorporate the more general changes into the main body of the Chapter and reserve unique issues for the appendix containing the requirements specific to air monitoring.

Proposed changes that get into the realm of the sampling will be addressed later in concert with the Field Measurements and Sampling Committee.

The QS Committee proposed that Mr. Glowacki draft the air testing section in Appendix D. This will be presented for discussion at the next NELAC meeting.

Attachment A

ACTION ITEMS
QUALITY SYSTEMS COMMITTEE
NOVEMBER 8-10, 1998

Item No.	Action Item	Date Completed
1.	Mr. Slayton to develop a form letter to send to commenters acknowledging receipt of their comment.	
2.	Mr. Slayton to find full citations for references to Section 5.9.4.2	
3.	Mr. Slayton will search Chapter 5 for the term MDL and replace with appropriate language. He will also eliminate references to 3.18 x MDL as a quantitation limit.	
4.	Prepare a template for commenters to use when submitting comments to the QS Committee.	
5.	A schedule needs to be proposed for the time to allot for the open meeting of the QS Committee at the next NELA Conference. Also, the meeting schedule of the QS Committee should be arranged so that Committee participants can attend the Onsite Assessment Committee meeting and the Field Sampling Committee meeting.	
6.	Discuss for Section 5.9.4.2.2.f.ii that the requirements should specify exceedance of a maximum regulatory standard because in some cases there may be a minimum standard as well as a higher maximum level as in the case of drinking water trigger levels for SOCs.	
7.	Mr. Porterfield will draft language, for Section 5.10.2.1, to address performing initial demonstration of capability on tests that cannot be spiked.	
8	Check the definition of calibration standard in the QAMS reference. If definition is not acceptable, then a new one will be developed.	
9.	Discuss Mr. Glowacki's definition of method blank.	
10.	Address IDOC regarding all media etc., how universal does this requirement need to be.	

Item No.	Action Item	Date Completed
11.	Create a second parking lot for the new set of written comments and assign them to Committee participants. The comments should be numbered to identify which QS Committee participant is assigned to review what comments.	
12.	Incorporate changes into Chapter 5 by the November 20th deadline for submitting chapters for publication for the next NELAC Conference.	
13.	Determine how to procedurally coordinate efforts of the QS Committee with the Field Measurements and Sampling Committee (and other committees as necessary).	
14.	Next QS Committee meeting will be a teleconference on November 12th. Preliminary agenda is to finalize the calibration and detection sections, address editorial changes from the air monitoring subcommittee, and respond to additional written comments.	

Attachment B

PARTICIPANTS
QUALITY SYSTEMS COMMITTEE
NOVEMBER 8-10, 1998

Name	Affiliation	Phone Numbers
Mr. Joe Slayton	USEPA, Region III, OASQA	T: 410-573-2653 F: 410-573-2698 E: slayton.joe@epamail.epa.gov
Ms. Mary K. Bruch	Mary Bruch Micro Reg. Inc.	T: 703- 589-1514 F: 703- 779-0267 E:
Mr. Raymond J. Frederici (Absent)	Recra Labnet - Chicago	T: 708-534-5200 F: 708-534-5211 E: frederir@recra.com
Mr. Clifford R. Glowacki	Ashland Chemical Company	T: 614-790-3482 F: 614-790-4294 E: cglowacki@ashland.com
Ms. Sylvia S. Labie (Board Liaison)	Florida Department of Environmental Protection	T: 904-488-2796 F: 904-922-4614 E: labie_s@dep.state.fl.us
Mr. David Mendenhall	Utah Department of Health	T: 801-584-8470 F: 801-584-8501 E: dmendenh@doh.state.ut.us
Ms. Sheila Meyers	Texas Natural Resource Conservation Commission	T: 512-239-0425 F: 512-239-6307 E: smeyers@tnrcc.state.tx.us
Mr. Jeff Nielson	City of Tallahassee Water Quality Division	T: 850-891-1232 F: 850-891-1062 E: nielsenj@mail.ci.tlh.fl.us
Mr. Donivan R. Porterfield	Los Alamos National Laboratory	T: 505-667-4710 F: 505-665-5982 E: dporterfield@lani.gov
Mr. Scott D. Siders	Illinois Environmental Protection Agency	T: 217-785-5163 F: 217-524-0944 E: epa6113@epa.state.il.us
Dr. Fred Siegelman	US EPA, QAD	T: 202-564-5173 F: 202-564-2441 E: siegelman.frederic@epamail.epa.gov
Mr. Mike Cross (Contractor Support)	Research Triangle Institute	T: 202-728-2045 F: 202-728-2095 E: myc@rti.org

Attachment C

PARKING LOT ITEMS/ISSUES
QUALITY SYSTEMS COMMITTEE
NOVEMBER 8-10, 1998

Parking Lot Items/Issues as of November 12, 1998. Items/issues will remain in the Parking Lot until they are completed.

1. Items for the NELAC Board to be forwarded by Ms. Labie

Query the reason for requiring written responses for each set of comments

Request addressing comments in chronological (first come first serve) order and QS approach:

- Short note acknowledging receipt and processing will be developed and routinely sent to commentors.
- Indicate that we prefer electronic format and specify format.
- Add section to routine QS meeting Agenda and associated minutes dealing with comments (which should serve as a log) and show status of whether discussed or not.

NELAC Interim Meeting and Conference Agendas: need to separate QS and On-Site committees so times do no overlap (at least 1/2 day without overlap). Ideally we suggest that the entire conference needs to be sequential for the standard setting committees.

Outreach for small labs - what is being done? We fear they do not have the resources or time to attend committee meetings, interim meetings or the conferences. How can we help assure that they are involved with the NELAC process?

Should there be a breakout session at the interim to brief the whole conference on the change to 17025 - educational to present the evolution from ISO 25 - should have no vested interest in NELAC - preferably one of the authors.

QS requests that a struck-through/underlined version of the QS chapter be available so that all other committees understand what changes are being made - ensures whether or not another committees (and all concern parties) will be aware of changes being made so they can more easily determine if the changes are of concern. In addition, this will help serve as a corporate record for the committees directly involved.

2. Air Appendix

The Air Analysis Workgroup has a number of editorial changes which were deferred from the November 8-10, 1998 QS Committee meeting because of lack of time. These items will be discussed at that time.

3. On-Going Issues

In 5.1 Scope (section b, 2nd item): "If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met (See the supplemental accreditation requirements in Section 1.9.2)." What if the standards are not the same and one does not appear to be obviously "more stringent"?
[note: one thought is that perhaps this should not be a major issue given that the two standards are probably of equal merit]

In addition, changes to the standard will be proposed at the January 1999 Interim Meeting, which will no longer specify the MDL (40 CFR Part 136) procedure be employed unless it is mandated by the test method or applicable regulation.

5. Revisit the Microbiology Appendix: Need for maintaining pure cultures of bacteria

With regard to testing of glassware washing technique and media for labs that only use media and soap which comes with manufacturer's certifications.

6. Proposed New Appendix

Appendix for listing of required records (all pulled into one table). Need to reach consensus on the table and the suggested introduction provided by D. Porterfield.

7. Continuous Monitors

Topic was briefly discussed at the Annapolis meeting (11/10/98) and it was decided that this topic may require its own appendix with associated special QC.

8. Action Items from the NELAC IV Conference.

This was a homework item and most of the work is completed but it has not been discussed.

9. **Initial Demonstration of Capability**:

Need to address an IDOC for tests for which you can not spike. Also, does IDOC need to be universal and address all medias?

10. Definitions

Method Blank (Cliff to provide)
Calibration Standard (Fred/Silkie to provide from QAMS reference)

11. Glossary

Changes necessary to be consistent with Program Policy and Structure proposal.

12. Matrix and Media

Suggestion has been made that the media definition should in turn be divided into a number of matrices. The committee has pulled into one file all items related to this issue (part of NELAC IV homework).

Attachment D

APPROACH FOR HANDLING COMMENTS
QUALITY SYSTEMS COMMITTEE
November 8-10, 1998

1. A form letter will be sent to each commentor notifying them of receipt of the comment and of the QS's approach to reviewing comments and associated updates to the standards.

2. QS will consider the comments in the order received.

3. A QS committee member will be designated as the lead on each set of the comments from each commentor. The QS member will provide written comments and will lead a discussion with the full committee on any proposed changes to the standards (including providing the proposed standard language).

4. Proposed changes to the standards will be captured in the QS meeting minutes which are posted on the NELAC World Web page.

5. All comments and written responses will be attached to QS meeting minutes.

6. No colors to be used in the comments nor in the response. Use double underlines for additions and strike-outs for removal of items.

7. All comments are to be provided in rich text format using the following the following table:

Comment ID #:	**Source of Comments (Name):**	**QS Lead on Response (Name):**	
Standard Rev. # SECTION# and QS Standard Narrative (To Filled In by Commentor)	**COMMENT to QS (To Be Filled In by Commentor)**	**QS Leader Provided Proposed Change (Commentor Leave Blank)**	**RATIONAL (from QS Leader) (Commentor Leave Blank)**

Attachment E

COMMENTS TO BE ADDRESSED
QUALITY SYSTEMS COMMITTEE
NOVEMBER 8-10, 1998

Listing of Comments Yet To Be Addressed:

C20: Virginia WEA NELAC Workgroup Comments (Sept. 30, 1998)

[QS Committee will review the first 9 pages of "major concerns" as a group and will select leaders (about 3 pages for each committee member)**]**

ISO 17025

[QS Committee will not comment as a committee, but QS members are welcome to comment as individual-indicating that their comments do not necessarily represent those of the QS committee. The QS Committee plans to schedule a meeting with invited experts on the proposed 17025 to highlight the differences between Guide 25 and the proposed standard, as well as well, to explain the vision/goals of the new standard. **]**

C21 Catalyst

[QS Committee will review comments on Matrix Spike- MSD as a group**]**

Attachment F

COMMENTS ASSIGNED TO QS COMMITTEE
PARTICIPANTS AND RESPONSES
QUALITY SYSTEMS COMMITTEE
NOVEMBER 8-10, 1998

NELAC QS COMMITTEE HOMEWORK ASSIGNMENTS AND RESPONSES
HOMEWORK ASSIGNMENTS

Comment Number	Commentor
C1	Mary Boatman (Texas TNRCC)
C2	Aquatech
C3	Water Envir. Federation
C4	Eastman Chemical Co. (Texas Div.)
C5	Carl Kircher (?)
C5B	Suburban Water Testing Lab
C6	Quanterra
C7	Castaic Lake Water Agency
C8	Bruce De Grazia (Assist. Deputy Under Secretary of Defense)
C9	David Brymer, TNRCC
C10	Silky Labie
C11	Barbara Hill (Waste Management)
C12	Jackie Saple
C13	J.R. Hall (?)
C14	Kodak
C15	Carl Kircher (2nd set of comments)

Mr Slayton will address comments contained in an October 5, 1998 Email from Ms. Cindy Dingman and Mr. Thomas McAninch (Eastman).

HOMEWORK

November 3, 1998

Homework Assignment

TO: Joe Slayton and Mike Cross

Aquatech
dated 7/17/98 from John and June Brien and laboratory
Comments by Mary K. Bruch, Nelac Quality Systems Committee

1. Section 5.9.3.a

This statement could be worded in a better way. I suggest a revision to read as follows: ...shall be used only for calibration, unless it can be demonstrated that their use as reference standards has not invalidated their further use in calibration.

The implication that an invalid reference can then be used for calibration is derived from a misreading of the statement as it is now written. The intention of the statement is exactly the opposite.

Section 4.1.1.1.

The Standard attempts to state that the Quality Assurance Officer is independent of the test(s) being run and has access to the higher levels of management. If the QA officer must sometimes function as a supervisor or even directly in tests, then the QA officer function must be temporarily transferred to someone else. In the Standard, the laboratory director is specified as, However named; meaning that the other titles listed, such as laboratory supervisor or technical director, can be the person in charge. As long as there are two people, the designated QA officer for a study can switch to technical supervisor or other designation for a specific test.

The one-person lab must deal with some outside source for QA support.

5.4.2.g.2
Removal from business operations is intended to relieve laboratory personnel of potential conflict-of-interest. The reporting of findings to the laboratory management is intended to eliminate possible pressures or conflicts in the performance of the test for the QA officer.

HOMEWORK

Comment review
Co: Kodak Chemical Co.
from: DV Zahnisser
Date: 8/20/98

Kodak Chemical Co. Issue (summarized)	QS response
Opening unnumbered issue: " the committee eventually has to come up with a term for "3.18 times MDL". Language provided for a "qualitative reporting limit" QRL.	Current revisions in 5.9.4.3 d addresses this concern. The committee's revision of detection and sensitivity addresses this issue **No further action by the committee is needed.**
5.9.4.2.1.c "Analytical Balances are a different type of equipement that is much less prone to deviation that ovens, refrigerators, or incubators." Suggested that balances be checked once per week rather than daily.	**Committee review requested.** This comment may have merit if the balance is not used for preparing reagents and standards. The daily calibration checks link the traceablility of standards to national weights and measures. The committee should consider allowance for weekly checks if the balance is not used for preparation of standards and reagents, and require checks whenever standards and reagents are weighed on the balance.
D.1.1.a Alternative text for the section was provided. "Method blanks shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method....."	**Committee Review requested** Revised language should be considered for incorporation into the standard.
D.1.4.c The commentor believes this section is redundant.	**No further action by committee needed.** The committee already deleted this section because it was redundant.
D.1.4.d Commentor is concerned that many laboratories routinely perform analyses far above the MDL and the the requirment for performing an annual MDL is not really relevent. Revised language was submitted.	**Committee review requested.** Revised language that was submitted should be considered for incorporation into the standard.

Comment review
Co: Eastman Chemical Co.
from: Tom McAninch
Date: ?

Note: Comments were submitted based on the May 98 Draft standards published prior to NELAC 4.

Eastman Chemical Co. Issue (summarized)	QS response
5.1.d "I support this addition to the standard. If a project/permit requirement does not require as stringent quality control as is required by NELAC, the lab must have the option of providing what the project/permit requires, but no more.	**No specific action by committee is needed.** Committee actions to address performance based measurement systems (PBMS) and EMMC consensus comments will also address this issue.
5.6.2.c.3 Comment on Analyst Training. "The addition of sub-paragraphs I, ii, iii, iv are a good addition to the standard.	**No action by committee needed**
5.9.3 Eastman believes the last sentence of paragraph a, paragraph b, and paragraph c states the same thing.	**No action by committee needed.** These paragraphs have three separate meanings and are not redundant a) refers to requirements the calibration body must use traceable reference standard when available b) refers to a requirement to have a program of calibration and verification for reference standards and c) requires the traceability of reference standards.
5.9.4.3.d Instrument calibration. " I am disappointed that the QS Committee failed to incorporate the Friedman/Haeberer proposal to allow one-sided calibrations in instances where reporting a "greater than" or a "less than" value is sufficient. ....." "QS also failed to incorporate the Friedman/Haeberer recommendation to allow the lab more flexibility to determine the number of calibration points to use. ....." However, under 5.1.d addition, if a permit writer agreed to the arrangement, I guess it would be allowed under the "less stringent" provision."	**Committee review recommended** The committee did not address this issue based on contradictions of feedback form the EPA. A consensus EMMC position will provide the committee the guidance to modify chapter 5. However, Eastman answered its own concern, by demonstrating a mechanism in the standard to use less stringent standards with appropriate approval and documentation.

5.9.4.4.2.c Continuing Calibration "At last! A lab is allowed to perform routine corrective action before having to construct a new calibration curve. This is badly needed change. However, the same language should be added to the initial calibration verification."	**Committee review requested** I disagree with recommendation. The corrective action language for continuing calibration is not applicable for the initial calibration. However, the committee may wish to consider alternative measures.
5.12.4 Although there are definitions for chain of custody and evidentiary chain of custody, the standard does not distinguish the difference between the two.	**Committee review requested** The standard does not clearly distinguish the difference between chain of custody (minimum custody required) from evidentiary custody. The committee should consider establishing the minimum custody requirements when evidentiary custody is not needed.
5.12.4.1.c "Thank you for recognizing that the customers/permit writers determine the content of reports....."	**No action by committee needed**
5.13.b At last, Ch 5 recognizes that customers/permit writers determine the content of reports....."	**No action by committee needed**
Ending unnumbered issue: "3.18 times MDL".	**No further action by the committee is needed.** Current revisions in 5.9.4.3 d addresses this concern. The committee's revision of detection and sensitivity addresses this issue

HOMEWORK C5

SECTION	COMMENT	REVISED SECTION	RATIONAL
5.9.4.4.1 a) When an initial calibration curve is not ~~run~~ established on the day of analysis, the integrity of the initial calibration curve shall be verified on each day of use (or 24 hour period). The analysis of a blank and a standard at the method defined concentration or a mid-level concentration may be used to verify the calibration if criteria are not included in the test method.	"…by analyzing a blank and a standard…" Is this standard a calibration standard, a reference standard, a quality control sample, or the lab's choice?	**No revision**	The label placed on the "standard" is unimportant. The important part of the section is associated with the concentration of the ""standard""
5.9.4.4.2 Continuing Calibration Verification Additional standards shall be analyzed after the initial calibration curve or the integrity of the initial calibration curve (see 5.9.4.3.a or 5.9.4.4.1 above) has been accepted.	Same question as above for the "Additional standards…"	**No revision**	Same as above
	Suggested addition: "Selectivity D.1.7 d) To validate selectivity of other tests, the laboratory should document spectral wavelengths used, quantification mass ions, electrodes used, and immunoassay enzymes (manufacturer and lot#), as applicable.	Add "D.1.7.1 The laboratory will document the selectivity of all test methods and that these criteria are met each day of operation. For example:	Expands selectivity to include all test methods, documents known interferences, and makes interference information available to client.
	Suggested addition: "Selecivity D.3.7 c) The laboratory shall document the manufacturer and lot number of enzymes used in immunoassay techniques and DNA sequences and labels used in polymerase chain reaction techniques.	No revision	Covered above

HOMEWORK

Quanterra Comments

The reference numbers in the body of the Quanterra letter reflect the number of the chapter prior to the 7/2/98 revision. Some wording was changed at the San Antonio meeting. Each reference point from the letter is addressed:

5.9.4.2.1 d]
From the Changes to Quality System(Revision-2 9/26/98) As a result of a comments/questions from the Illinois EPA concerning microliter syringes:

5.9.4.2.1.d. Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on a monthly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

5.9.4.3 b] 1] i] and ii]
From the Changes to Quality System(Revision 2 9/26/98) As a result of comments from EPA (EMMC and program offices) as well as numerous concerns involving the great detail in section 5.9.4.3 Instrument Calibrations:

A re-write stresses establishing a QS framework which ensures that the data will be of knows quality , appropriate for a given regulation or decision, while allow allowing laboratories to select the appropriate techniques.

5.9.4.3 d]
Comment: The requirements of this chapter will be the minimum and take precedence over less stringent method requirements. The more relaxed SW-846 will not be allowed.

5.11.2
Comment: Making the sample acceptance policy available to the laboratory's sample collection personnel is obvious. Making the sample acceptance policy available to the laboratory's client would meet the requirement since the client may use their own personnel or a subcontractor to collect samples. In either case the collectors would be acting as agents for the client and would be the clients responsibility.

5.12.2
Comment: This requirement is, that a plan exists "to ensure that records are maintained or transferred...in the event a laboratory transfers ownership...". Meeting this requirement would be based on the fact that there is a plan. The reasonableness of the plan is not a concern as this requirement is written since no standards are described in the chapter. Specifically addressing the concern of who would pay for the activity in the event a laboratory goes out of business would be a critical point of a plan.

5.12.4 c]
Comment: The point in question is whether "c] The COC records shall identify all individuals who physical handled individual samples." should be a requirement. This level of security is not for routine samples but only those that are specifically requested to follow legal or evidentiary procedures. When a laboratory accepts sample of this type [legal or evidentiary custody] the requirements of this chapter come into affect and so "5.12.4. c" is required for any legal or evidentiary custody samples. It does seem to be industry standard to keep a record of anyone who takes custody of this type of sample during the process. The final judge of the effectiveness of the laboratory's legal or evidentiary custody procedure would be the court system as results are accepted or rejected as evidence. The laboratory would have a chance to explain it's procedures and document it's practices. Not following the requirements of the oversight body [NELAP] or industry standard would put the laboratory on somewhat shaky ground.

5.14
This was corrected in the 7/2/98 revision of the chapter to read:

5.14 SUBCONTRACTING ANALYTICAL SAMPLES

a) The laboratory shall advise the client in writing of its intention to sub-contract any portion of the testing to another party.

b) Where a laboratory sub-contracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed.

c) The laboratory shall retain records demonstrating that the above requirements have been met.

Appendix B - Definitions

quantitation limits

Comment: The definition indicates that the quantitation is 3.18 times the MDL, by convention. That implies that it is not a set factor but one agreed to by participants. It also implies [to me] that there may be exceptions. Listing every exception is not possible but a phrase like "when appropriate" is too open ended. The suggested wording seems appropriate: "..... Quantitation limit, for the purposes of NELAC, is defined as 3.18 times the MDL, by convention. This convention is held if the value is at or above the lowest calibration standard. If this value is not at or above the calibration standard, the quantitation limit becomes, effectively the lowest calibration standard." (see 5.9.4.3d]).

Appendix D - Essential Quality Control Requirements

D.1.1 a] 1] Chemical testing, positive and negative controls, negative controls, Method blanks.

Current wording:

1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

   i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch and
   ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

   Each sample in the affected batch must be assessed against the above criteria to determine if the sample datum is acceptable. Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

Comment: As written the requirement in i] seems excessive. The definition of MDL indicates that values below it are difficult to differentiate from zero.

Using the regulatory limits as an action level is a problem for the reasons stated by Quanterra and also since the regulatory limits are so difficult to identify [which program, agency, permit, etc.].

The realistic approach, from the laboratory's perspective, seems to require investigation and corrective action when blank results exceed the quantitation limit as Quanterra recommended. It would be prudent, however, to begin looking at the system when the blank results exceed the MDL and evaluate for trends at that point. That may be a valid negotiated position to raise the action level from 1/3 MDL [as it is now] to the MDL.

HOMEWORK

Castaic Lake Water Agency
Ad Hoc Laboratory Accreditation Work Group

Comments submitted by: Miriam Cardenas & David Eugene Kimbrough (Co-chairs of Ad Hoc)
Comments submitted to: Dr. George Kulasingam (ELAP)

COMMENT #	COMMENT	QS RESPONSES
1) page 1 of 5	"The Data Quality Objectives (DQOs) of the various end users of data generated by NELAC accredited laboratories are not adequately addressed in this Standard. This goal is missing througjout the NELAC standards in general, and in Chapter 5 in particular. Without this aspect, the checklists and the Chapter 5 Standard miss the mark."	Chapter 5 defines DQOs necessary for a laboratory to be NELAC accredited. It is not within the scope or intent of NELAC to address the DQO's of the various end users of data generated by NELAC accredited laboratories. As stated in your comment, DQO's ***are*** determined by the end users (not NELAC) and can be site, project and program specific. The scope of Chapter 5 is to provide a consensus of ***minimum*** standards necessary for the regulated chemical and biochemical measurements, thus providing a baseline of essential quality control procedures.
2) page 1 of 5	"There are no ranking of deficiencies. The Standard must clearly distinguish between recommended Good Laboratory Practices and mandatory and enforceable requirements. For example item 2.1 asks 'Does the purge and trap system contain a purging device, trap and desorber" and item 3.19 asks " Is the standard with the lowest concentration run first"?. Is a no response for these two questions of equal importance, i.e. is running standards out of order of equal importance to not having a purge and trap for Method 502.2 or 602?"	*Comment should be taken up with the On-Site Assessment Committee.*
3) page 2 of 5	"The proposed auditor checklist are limited to only two instruments, purge and trap gas chromatography (GC) and gas chromatography-mass spectroscopy (GC-MS). If ELAP is required to use NELAC checklist by January 1999, then there is a serious deficiency in the number of check list. The existing lists are also far too long, the questions are all leading and not methods specific (e.g. 502.2, 602, or 8000B etc.). Further, how Are the Performance Based Method Systems (PBMS) methods to be evaluated using this form? A laboratory with more than one purge and trap method might answer differently for the same item."	*Comment should be taken up with the On-site Assessment Committee.*

4) page 2 of 5	"The main Quality Systems checklist is unbelievably long (72 pages) and would require many days of an auditors time on-site to answer, to say nothing of all of the additional instrument based checklist." "Many of the questions are vague or ambiguous and highly subjective. For example, item 5.6.1 asks, "Does the laboratory have suffieient personnel, having the necessary education, training, technical knowledge and experience for their asssigned functions?" What is the criterion for making this assessment? Since no criterion is offered, it is up to each laboratory assessor to make a subjective asssessment. There many similar questions through out this checklist."	The content of Quality Systems was supported and voted upon by the majority of the stakeholders with direct influence from the non-voting members. The length of the section directly relates to providing specific standards in order to minimize ambiguity. Section 5.6.1 - directly from ISO 25 states: " <u>The laboratory shall have sufficient personnel, having the necesssary education, training, technical knowledge and experience for their assigned function."</u> This deficiency would be cited and supported directly by non-arbitrary laboratory deficiencies (e.g., lack of knowledge of SOPs, improper calibration procedures, lack of proper documentation, etc.) which would be a indicated on the assessor's checklist. (See On-Site Assessemment checklist) Problems documented within the lab by the assessor will clearly identify insufficiencies in personnel qualifications, training, experience deemed essential to quality assurance/quality control tasks necessary according to quality objectives set forth by the NELAC standards.
5) page 1 or 5	"Other questions are impossibly broad and unenforceable. Item 5.8.b asks 'Is all equipment properly maintained, inspected and cleaned?' Again no standards are offered as to what is "proper" in terms of maintenance, inspected and cleaned?" Once more the assessment is entirely subjective. Further, there can be dozens of pieces of equipment in even a small laboratory, which ones are being referred to. How can a Yes or No answer this questions for one instrument much less a laboratory full of instruments. Item 5.9.4.4.1.b asks 'If the initial calibration verification fails, is the analysis procedure stopped and evaluated?' Even a small laboratory might have a dozen methods requiring an initial calibration curve. This checklist is full of such questions."	Item 5.8.b is taken directly out of ISO 25 and applies to all equipment that supports measurements of regulated environmental measurements. Records that must be maintained 5.8 e) 1- 9 are standards describing what must be documented to demonstrate that equipment is properly maintained. The standard focuses upon the laboratory ***having*** maintenance procedures that demonstrate that equipment is properly maintained, ***not*** what these prodecures should be. As these would be too numerous to name.

6) page 2 or 5	"The quality systems standards are a "one size fits all' standard. We believe that there must be a procedure for customizing requirements to fit both the large commercial laboratory and the smaller municipal laboratory. For example the requirements for internal audits. In a two or three person laboratory, how could this be performed? The supervisor may well be the QA officer as well as an instrumental analyst. Item 5.5.3.1 asks "are clients notified immediately, in writing, when their work is affected by the finding from an internal audit?" Many municipal laboratories have only on 'client'. There are many other examples of organizational requirements that assume the existence of a large and complex structure. The level of documentation on personnel training and qualification, sample tracking, supervision, disposal, LIMS usage, etc is ridiculously out of proportion for a municipal laboratories with only a few employees."	Again, the quality systems standards are a consensus of minimum quality assurance protocols necessary to support the laboratory's quality system and therefore must be independent of number of staff. It has been determined that internal audits are just as important for smaller labs as larger. For a two or three person laboratory, people could be selected to perform a laboratory audit over data not generated by them. Also, please note what the standard says: "***...whenever possible***, independent of the activity to be audited." *I Do not understand the relevance of 5.5.3.1 and the statement that municipal laboratories having only one client.* QS finds number of employees irrelevant to essential doucmentation required.
7) page 2 of 5	"There is confusion in the Standard between method requirements and quality systems requirements that must be clarified in order to provide for effective implementation. The documents do not afford any evaluation of many important method specific requirements (e.g. GC-MS tuning requirements, pesticide brake down product testing) that are crucial to effective evaluation of laboratory performance. Conversely, there are many overly broad requirements such as MDL studies for alkalinity which do not contribute to the improvement or evaluation of laboratory performance. As well, there are arbitrary quality control requirements that do not serve the data quality objectives of the end user, the laboratory or the laboratory assessors, such as the 15% continuing calibration verification requirements."	Again, Quality Systems Standards are the minimum standards that must be met. If more stringent requirements are detailed in a method, the more stringent requirements apply. If no standard or less stringent standards are listed in a method, the lab must meet standards as designated in the NELAC standards. It is not necessary for NELAC standards to reiterate specific requirements in all the methods. As you stated, these are "method specific" and already detailed in each of the methods. This would force the document to become twice as long with no added value. Earlier comments by your Ad Hoc committee stated that the standard was already too long. *Talk to QS group about alkalinity*. MDL procedures are being reworked. QA committee is currently rewriting chapter 5 calibration procedures.
Chapter 5 NELAC written comments		

1) page 4 of 5	5.4.2 Many labortories are simply too small to have a QA officer who is "Independent of laboratory operation."	*I think our committee should add some language for one person laboratories. Note: ISO 25 and DIS 17025 state nothing of a "QA officer" or designee and independence from laboratory operations. I agree with the comment. This is not practicle for a large percentage of small, and municpal laboratories.* *See section 4.2 in ISO 25 and 4.1.4 in DIS 17025.* Suggested revision: 2) have functions independent from laboratory operations for which they have quality assurance oversight **where possible** ; *NOTE: We have already allowed language as **"whenever possible"** (5.5.3.1) when referring to the internal laboratory audits. So we have essentially acknowledged a laboratory limitation.*
2) page 4 of 5	5.4.2 Is past experience in auditing considered training? How does one become trained and qualified as an internal auditor? What are the standards for adequate internal audits? How will it be enforced without standards	Yes, it could be. On the job training. Classes. *(We may need to re-think this) Can't require something not readily available.*) *Don't know* ???
3) page 4 of 5	"Can QA officer be considered management and take on some of these responsibilities? e g., signing current QA documentation, etc? Again many small laboratories do not have such elaborate structures."	Yes. Please refer to the standard 5.4.2 g) which states that the quality assurance officer may also be the technical director of deputy technical director.
4) page 4 of 5	"5.5.3.5 B Is data not considered unreportable if out of control? If we do use qualifiers, what qualifiers will be used? Do they need to be established all labs to use. B=Blank contamination, H=holding time expired, etc. Many data qualifiers are specified by data users."	NELAC criteria specifies that data "out of control" be qualified. How laboratories qualify may vary. All reports however, must be clear about the use of qualifier(s) and definitions so that the data users may be aware of possible limitations of data usage.
5) page 4 of 5	"5.11.1 Does the Sample Tracking have to be hand-logged, or can it become part of our LIMS system and be electronicaly based?"	Section 5.11.3 d) states: "The laboratory shall utilize a permanent, sequential log, such as a log book or **electronic record**, to document receipt of all sample container."
6) page 4 of 5	"5.7 In many smaller laboratories these have to be combined in one way or another. Can any of these areas be combined?" ***This comment refers to work areas and separation.***	Work areas combined in the same room must have "effective separation". Effective separation can be demonstrated by the documentation of blanks used throughout the sample preparation and measurement process to verify non-contaminating conditions in the laboratory.

7) page 4 of 5	5.9.4.3.a CCV shall be within 15% of the true valuse unless the lab can demonstrate through historical data that wider limits are applicable. What is considered historical data? What is the criterion for assessing what is "applicable?' Must all data prior to establishing that this is an historical fact be qualified? Many methods require a low-level CCV and mid-range CCV. The low level CCV generaly has a wider acceptance range than 15% sometimes as low as 50% window of the true value, as in the TOC low level Ccv in the ICR. Can we get some wider ranges here, or adhere to the methods iven if the method has LESS straingent protocols? 15% seems very arbitrary. What is its basis? Again the data quality objectives of the data user should be paramount."	Section 5.9.4.3 is being revised.
8) page 4 of 5	5.9.4.3.b, 5.9.4.4, 5.9.4.4.2 What is the point setting up very specific requirements that do not ally to any specific method that have no basis in the data quality objectives of the end data user or any specific method or program requirement?	As stated above, these sections are being revised.
9) page 4 of 5	5.10.2.1 Could this be the same resource, since many SOPs include methods?	*Not sure I understand the question*.
10) page 4 of 5	5.10.2.1 Is any sort of approval required before implementing PBMS? What happens if the laboratory assessor decides that the PBMS is not adequate during an audit? Will the data generated be invalidated? Will the laboratory be decertified for that analyte?	No approval will be required to initiate PBMS for measurements that allow it's use under regulations. The laboratory however will have to show all necessary documentation necessary for it's implementation during the on-site assessment. An assessor cannot arbitrarily decide that the PBMS is not adequate. This decision, which will be obvious to both the laboratory and the assessor, will be measured by performance standards, (which will be specified) meeting or not meeting stated criteria. Data generated and reported out for any method used, which falls short of required performance standards and does not get qualified, will be invalid. A laboratory reporting out suspect data without qualifiers will be decertified for that analyte.

11) 5.12.3.1	Sample Handling. A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to...g) Equipment receipt, use specification, operation conditions and preventative maintenance; h) Calibration criteria, frequency and acceptance criteria; I) Data and statistical calculation, review, confirmation, interpretation, assessment and reporting conventions; J) Method performance criteria including expected quality control requirements; K Quality control protocols and assessment; l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; m) All automated sample handling systems; n) Records storage and retention; and o) Disposal of hazardous sample including the date of sample or subsample disposal and name of the responsible person. **Questions & Comments: this is either redundant with other sections (record keeping etc) or wild over-kill for sample handling records.**	QS does not agree with the comment. Section 5.12.3.1 is in fact a unique, complete and essential list of records, not duplicated elsewhere in the standards.
12) page 5 of 5	5.13 Report requirements are set by the data user. In California the Department of Health Services has its set of required forms.	QS believes that section 5.13 details minimum reporting criteria neccessary to support the interpretation of the test results. However, the reporting format may vary.
13) page 5 of 5	Appendix D There is not mention of Laboratory Control Samples until the Appendix. There should be some reference to them in the standard perhaps where the CCV's are to avoid confusion.	QS believes that the the laboratory control sample is one of several controls. We have choosen for simplicity to separate the quality control samples from calibration procedures, which are being revised.

14) page 5 of 5	Appendix D.D1 2 What should a lab do if a matrix spike is "in" and the duplicate is "out"? What if the concentration in the sample is so high that the percent recovery is very low?	The lab should initiate some kind of corrective action. Possibilities include: repeating the analysis; verifying acceptance of the laboratory control sample, compare analyte concentration results. Repeating preparation of sample for analyst reproducability etc.. If corrective action does not reveal nature of problem and the error not corrected, the data will have to be qualified as indicatied on the report sheet. I'm guessing that the question is, "what do you do if the sample concentration is so high that it interferes with MS recovery" However, the question is still somewhat incomplete. (*But I will take a stab)* First of all, I'm not sure if you are referring to matrix interferences. Matrix interference that can be verified by laboratory procedures. If so a clean up method may help. If the sample concentration is so high, chances are a dilution, or several may have to be used to get the concentration between the calibration standards for reporting purposes. If dilutions are made to the sample, obviously the MS will be diluted as well. For a semi-volatile method, repeating the analysis with higher concentration will set you back at square one but it is an option. Volatiles could be repeated quickly with a higher concentration of MS prior to dilution. *The scope of this comment is too broad to go into here.*
15) page 5 of 5	D.I.7b If an ample from a new site for THMs is analyzed and it is expected to be a certain level, do we need to confirm just because that particular site has not been tested before?	For positive results, D.1.7 states: "Confirmation is required unless stipulated in writing by the client."
16) page 5 of 5	PBMS rules??? "at least 4 replicates should be prepared and anlyzed independently. Is this on 4 separate days and prepared four separate time? How is PQL or MDL determined in this section. Frequency of performance of Initial demonstration = how does a lab know the frequency required?	No. QS is revising language on MDL. Also refer to EMMC. This is a one time procedure per analyst per method.

HOMEWORK

homework changes addressing D.1.1. as follows:

A blank is contaminated if
i) the contamination exceeds the MDL and
ii) (formerli i)
iii) (formerly ii)

HOMEWORK

Homework #C9

Issue from David Brymer, TNRCC, 512-239-1725

Issue regards 5.9.4.4.2.a (Continuing Calibration Verification)
which currently reads:

> These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be the standards used in the original calibration curve or standards from another source. The frequency shall be increased if the instrument consistently drifts outside acceptance criteria before the next calibration.

Comments:

> Mr. Brymer's concern seems to be in the area of continuous monitoring systems and how the language above would impact the frequency with which these systems are subject to a CCV. If a continuous monitoring system were to acquire a sample/ data point every 5 minutes then a CCV would be required on the order of every 2 hours. This when such systems are currently allowed to run CCVs once per day or even once per week.

Suggestion:

> In addressing 5.9.4 in Annapolis we should address whether the CCV frequency should be left up to the respective method or if we should specify some minimal frequency. And in the case of the latter whether we should give the continuous monitoring community some specific leeway on this issue.

HOMEWORK

0/19/98

I would agree with Silky's proposed changes to 5.11.3.a.2 with the following modifications:

Confirmatory preservation checks are not required by the laboratory when:

i) samples are properly collected, following written standard operating procedures, and are immediately hand delivered to the laboratory by laboratory personnel within one hour of sample collection,

ii) proper sample preservation, as specified by a standard operating procedure or test method, is known to have occurred and can be verified to have occurred by review of documentation or records; and

iii) analysis or sample preparation must begin within the specified holding time.

Scott

Obviously more "late" homework to come.

In looking at the standards it appears to me that the analytical records called for in 5.12.3.3 stand alone and are not addressed elsewhere in the chapter.

Scott

I would make the following changes to this section to address the issue at hand:

1. Add after the first use of "test method" the following:

(see 5.10.2.a.1)

2. Delete the following phrase:

"the test method has an extremely long list of components"

3. Delete the last sentence of D.1.1.b.4.

I feel these changes provide us what is needed as the essential requirements. If a client, project, mandated test method calls for what was specified in the last sentence then what we already have in D.1.1.b.4 should cover it (verify to an adequate degree that the analytical system if functioning properly and whether matrix effect is coming into play). Especially the part, "shall include permit specified analytes and other client requested components." I think having the last sentence included in D.1.1.b.4 with the changes I have suggested and the present langauge is not needed and is to prescriptive.

HOMEWORK

Response to comments from: Barbara Hill, Waste management

The comment requests text retained in Chapter 5 because they are in ISO standard and Chapter 5 needs to keep the text:

Request to retain text in section 5.4.2 e:

The ratio of supervisory to non supervisor personnel shall be such as to ensure adequate supervision.

Response: This sentence was retained in the July 2, 1998 Revision 9 version. This sentence appears in ISO 25 4.2 e). It does not appear in DIS 17025 4.14 g) which is, I believe the paragraph equivalent to ISO 25 4.2 e)

Request to retain text in section 5.9.3 c:

Where relevant, reference standards and measuring and testing equipment shall be subjected to in service checks between calibrations and verifications.

Response: This sentence was retained in the July 2, 1998 Revision 9 version. It appears at ISO 25 9.6. I did not find the same text in DIS 17025 which has

5.6.3.3 Intermediate checks

Checks needed to maintain confidence in the calibration status of reference, primary , transfer or working standards and reference materials shall be carried out according to defined procedures and schedules.

Request to retain text in section 5.13 a) 11:

measurements, examinations and derived results supported by tables, graphs, sketches and photographs as appropriate ..... to provide data.

Response: Most of this text was retained in the July 2, 1998 Revision 9 version.

The parenthetical phrase: (**such as failed quality control**) was not retained

the sentence: **Where relevant, include a description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data.** was not retained.

The text: **Where applicable** was not retained.

ISO 25 13.2 k matches the July 2, 1998 Revision 9 text: **measurements ..... failures identified.** I could not find identical text in DIS 17025.

I could not find the original source of the parenthetical phrase: (**such as failed quality control**)

The sentence: **Where relevant, include a description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data.** Is similar to 40 CFR § 160.185 (a) (11), the FIFRA GLP regulations.

The text: **Where applicable** deletion was an editorial change.

Recommendation: We do not need to make any change in our current draft to respond to this comment because we have retained the ISO 25 language the comment was asking for. However, if DIS 17025 in its current form replaces ISO 25, We may be asked to make some major changes.

HOMEWORK

C15

From: Cindy Dingman <cdingman2@hotmail.com>
To: R3MD1.R3CRL(SLAYTON-JOE)
Date: 10/5/98 2:49pm
Subject: Quality Systems

It is my understanding that for hazardous waste testing under the auspices of RCRA, that the laboratory seeking accreditation must meet all the requirements listed in general laboratory (NELAC Chapter 5), chemistry, (NELAC Chapter 5, Appendix D.1), the RCRA regulations (40CFR261), and the methods used in SW-846. Pat Mack from USEPA Region 9 stated that the current NELAC standards in Chapter 5 are for drinking water only and that the Standards for RCRA will not be discussed until after the July 1999 NELAC meeting. Any clarification on this matter would be greatly appreciated.

Thank you in-advance

Cindy Dingman
QA Officer
CalEPA
Dept. of Toxic Substances Control
Hazardous Materials Laboratory
(510) 540-2329
cdingman2@hotmail.com
cdingma@ix.netcom.com

Response: The Quality System Chapter 5 of NELAC is to apply to all the EPA's regulatory programs, i.e., this is the defined scope of NELAC.

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C16

From: "Mcaninch, Thomas W" <twmcan@eastman.com>
To: Joe Slayton <slayton.joe@epamail.epa.gov>
Dates: 10/5/98 9:39 am
Subject: NELAC & PBMS

I have had several questions from lab personnel concerning PBMS. I know QS has not yet adressed all the issues under PBMS, but if you have an answer for this, I would appreciate your thoughts. This may be an Accreditation Process question, but if so, I would appreciate your thoughts anyway.

I know there are provisions for adding analytes to your accreditation between accreditation certificate issuances, but has the question of a lab changing a method been addressed? What happens, under PBMS if a lab determines that it needs to modify their method for which they are accredited to meet the DQO's of a new project? Since this will probably not be uncommon, I presume there will be some procedure to allow this to happen within some reasonable constraints.

Response:

It is agreed that this is more an Accreditation Process question. From a QS Committee point of view our goal has been to delineate essential QA and QC procedures, which should help assure, measure and document data quality regardless of the analytical method system currently in place or the method selected (or developed) within such systems, e.g., even

under PBMS a laboratory would have to continue performing the QS standards (general items in the body of the chapter and specific listed in Appendix D.

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C1

From: Mary Boatman<MBOATMAN@tnrcc.state.tx.us>
To: R3MD1.R3CRL(SLAYTON, JOE)
Date: 7/3/98 10:32am
Subject Comments on Chapter 5

I am the person who conducts inspections of environmental laboratories in the State of Texas for the TNRCC. I was reading the NELAC standards approved July 1997 and I have a few comments. I attended the meeting this week and I don't believe any of the sections I am commenting on were a part of the changes made.

1. Should someone use the initial calibration or daily verification for quantitation? I was looking at section 5.9.4 and this is not clear. I do visit some laboratories that use the daily calibration for quantitation for GCMS analysis. I believe in S-846, method 8000, it does state that the initial calibration should be used for quantitation. I think that this should be made clear in the standards, if one method is preferred or required over the other.

Response: The QS committee is rewriting sections of the standard concerning calibration. We will consider clarification of what standard or calibration that is to be used for determining sample concentrations.

2. Section 5.10.5- Traceability of standards and reagents. A problem that I have with laboratories is that they do not indicate on the daily runlog which standard was used. As I read through this section, I see where the preparation is to be documented and traceable to NISTstandards through the certificate of analysis and the container that is used is to be labeled. However, there is nothing in the standard that I could find that would require documentation linking the calibration standards, the spiking standards, surrogate standards or the LCS used on a particular day to the NIST certificate. This will lead to man different modes of documentation or lack of documentation.

I tell people that I should be able to take the CCV from a particular day and trace through the paperwork back to the certificate. The bottle would not be available 2-5 years from now when this information may be necessary. I think the standard should include another section to provide this missing link.

Response: The QS standards regarding calibration are being revisited, however we will assure that traceability of calibration standards to a national standard reference material as in section 5.9.2 (Traceability of calibration). However, such traceability for surrogate standards and other spiking materials has not been considered essential and has not been included in the standard.

3. Section 5.11.3.d.6. The signature or initials of the data logger should be the original and not computer generated since anyone can use someone else's initials or signatures can be computer generated.
Response:

Section 5.11.3.d.6 allows for the electronic storage of signatures or initials. We will consider the addition that permits and other mandatory monitoring may require that such items be written/hard copy recorded. On Nov. 10 the QS Committee decided that your proposal would be overly restrictive for the QS standards but that EPA program requirements may address your concerns.

4. Section 5.12.3.3.f The same as in item 3.

Response: Section 5.12.3.3.f as does 5.12.3 (Laboratory Sample Tracking) does not exclude electronic storage of analysts signatures or other information**.** We will consider the addition that permits and other mandatory monitoring may

require that such items be written/hard copy recorded On Nov. 10 the QS Committee decided that your proposal would be overly restrictive for the QS standards but that EPA program requirements may address your concerns.

5. Section 5.12..3.3.f. The identification of the standards used in the analysis for traceability purposes should be included.

Response: With regard to 5.12.3.3 (Analytical Records), we will consider the addition of 5.12.3.3.g, Identify the source of the analytical standards with regard to traceability to a national standard.

6.Section 5.13. a.13. I would be concerned that an electronic identification of the person accepting responsibility could be forged easily.

Response: Section 5.13.a.13 (Laboratory Report Format) includes electronic identification of the persons accepting responsibility for the content of the certificate or report and date of issue. We will consider the addition that permits and other mandatory monitoring may require that such items be written/hard copy recorded On Nov. 10 the QS Committee decided that your proposal would be overly restrictive for the QS standards but that EPA program requirements may address your concerns.

7. Appendix- D- D.1.1 and D.1.2- As I read it, the matrix spike and spike duplicate can be added on an "open" batch basis. If a laboratory prepared 20 semivolatiles in a year, they would only need to do one matrix spike and spike duplicate during that year. I stress to people to used closed batches with matrix spikes prepared on the same day as the samples. In method 1664, this is how a batch is defined. Is it the intent of NELAC to allow open batches in this case?

Response: With regard to Matrix Spikes and batch requirements, a batch is defined as 1 to 20 samples , so that even if you only analyze one sample the laboratory would have to perform all the QC required of a batch, e.g., MS.

8. Section 5.9.4.3.c.2 This hat section states that the correlation coefficient shall be no less be no less than 0.995. This contradicts which allows a coefficient of 0.99 for organics.

Response. With regard to section 5.9.4.3.c.2 and correlation coefficients, this section is be totally rewritten.

Thank you for considering my comments.

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C17

Unknown Commenter???

1. There are several references in the NELAC standards that can be construed as precluding PBMS as it has been defined by the U.S. EPA because of notification requirements. This is largely because of an inconsistency between the field of testing as described in Chapter 5 acceptance limits and pass/fail criteria as established on a Program-matrix-analyte basis as per wording in Chapters 3 and 4 which describe the field of testing on a Program-method-basis. It is our understanding that it is the intent of Chapter 5 workgroup to adopt this from Chapter 2; this will be very beneficial to harmonizing the Chapter 5 guidelines with the goals and objectives of the PBMS. Clarifying this confusion will minimize the likelihood of separate PT's, separate on-site assessments, or that new certificates will be required in re-notification of intended changes in method /technology within the field of testing.

2. Past experiences with the States suggeststst that they will not hesitate over time to make "notification" mandatory or pre-notification on" to "submission for approval" and thereby compromise the basic precepts of PBMS.

POSSIBLE SOLUTION: (provided by commenter)

1.Promptly adopt the "Program -matrix-analyte basis language in Chapter 5, and work with Chapter 3 and 4 groups to obtain consistency on this basis throughout the standards.

2. Make language regarding notification of any changes in key "accreditation criteria,, 4.3.2 and analogous sections in Chapter 5 consistent with the understandings described above, and indicate that notification requirements, where they exist specific to the program, will not constitute advanced approval of changes made within the field of testing defined on a program-matrix-analyte basis.

3. Consider suggesting a form that can satisfy programs requiring notification/pre-notification for method changes that is simple, unambiguous and allows any proprietary issues to be identified by the laboratory.

1. Issue: Approval...

Response: Such notification is not currently required in the QS Chapter 5 standards.

2. More demanding State requirements.

Response: NELAC is to preclude states from having additional requirements over and above those in NELAC standards.

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C12

Note to "Silky" from Jackie Sample, DON at the July IV NELAC:

Please review the following as the ISO 25 language and intent appear changed:

* P.113, 5.4.2.e (Organization)

Response: Supervisor familiar with testing and calibration, the QS Committee has updated this section to be more consistent with ISO 25 (4.2.e).

* P.115, 5.5.3.1 (Internal Audits):

Response: We think that this and associated sections in the QS chapter (Internal audits, managerial review, audit review and performance audits) match well ISO 25 (5.2-5.6).

* P.119, 5.9.3.c (Reference Standards):

Response: We think that this section matches the intent of ISO 25 (9.2 and definitions in 3.12). We will consider however changing the term reference standards to the iso "reference material" (ISO 25, 3.11). On Nov. 10, 1998 the QS decided that the current NELAC standard equates the two terms and no additional changes to the standard are necessary.

* P. 128, 5.13 (11) (Laboratory Report Format and Content):

Response: We think this is consistent with ISO 25 (13.2.k), i.e., lab reports are to contain supporting material as tables, graphs, etc.

Section 5.6.2: "...Statistically identical results" is vague. Do you mean a t-test? If so, at what significance level? What alpha error? What beta error? I can imagine any number of statistical tests to yield different answers & different conclusions.

Response: This QS section delineates standards for how an analyst demonstrates they have sufficient experience and knowledge of the method and equipment necessary to begin performing analyses. Specially this section discusses the least desirable option (when performance testing or external quality control material are not available) in that the new analysts analyses the same material as the trained analyst. The QS Committee will consider changing the wording from "statistically identical results" (one proposal being "with comparable results").

ACTION ITEMS FROM NELAC IV (FROM ACTION ITEM TABLE FROM "SUMMARY OF THE QS COMMITTEE MEETING JUNE 29-30, 1998" AS WELL AS ADDITIONAL ITEMS FROM THE NARRATIVE OF THE MEETING SUMMARY. COMPILED BY MR. SLAYTON AND CIRCULATED TO QS 8/23/98)

From the Summary of the QS Committee Meeting (June 29-June 30, 1998) NELAC IV 8/23/98 "leftoff.wpd"

From the narrative:

Section 5.10.2.1.d. Method Validation. Need to work on per matrix/media, etc. and related topic Appendix C.1.b-Definition of Medium/matrix. **(Mr. Mendenhall)**

Section 5.9.4.3.d Instrument Calibration. To be addressed in detail. **(Continue as group effort)**

Appendix D.1.4.c. Method Detection Limit and Definition Limit of Detection. It was decided to leave it in as is and clarify it over the next year. **(Continue as group effort)**

Appendix D.5. Air Testing. (Subcommittee Cliff Glowacki, Susan Kilmer, Mike Poore, Gene Riley, Don Russel, and Hank Taylor). Four sections to review in detail. **(Mr. Glowacki is the point of contact)**

From the table at the end:

1. Committee to read glossary definitions of "medium" in order to decide wording changes (substitution of "medium" for "matrix") for Section 5.10.2.1. **(Mr. Mendenhall)**

2. Committee to decide if there is a need to exempt preservation checks in certain circumstances and to identify specific standards if required (Section 5.11.3). **(Ms. Myers)**

3. Committee to consider substituting "level of detection" for "limit of detection" in appendix B (Definitions). **(Continue as group effort)**

4. Committee to consider clarification of language in D.1.1 concerning blank contamination levels which are lower that method detection limit. **(Mr. Nielson)**

5. Committee to determine whether quantitively reported result values above the regulatory level should be bracketed by standards. **(Mr. Porterfield)**

6. Committee to consider the alternatives to the current MDL requirements and propose changes. **(Continue as a group effort)**

7. Committee to consider other approaches to calibration, and the merits of single point calibration curves and propose changes. **(Continue as a group effort)**

8. Committee to reconsider the time frame for spiking all components of a multi component method. **(Mr. Siders)**

9. Input form the Air Subcommittee must be completed. **(As above)**

10. Incorporation of Air Subcommittee recommendation. **(As above)**

11. Response to Suburban Water Testing Laboratory, Inc. Letter. **(Addressed)**

12. Consider comments received from April 15 through July 2, 1998. **(To be addressed as indicated in Item 2)**

NELAC QS COMMITTEE HOMEWORK ASSIGNMENTS AND RESPONSES

HOMEWORK ASSIGNMENTS

Comment Number	Commenter
C1	Mary Boatman (Texas TNRCC)
C2	Aquatech
C3	Water Envir. Federation
C4	Eastman Chemical Co. (Texas Div.)
C5	Carl Kircher (?)
C5B	Suburban Water Testing Lab
C6	Quanterra
C7	Castaic Lake Water Agency
C8	Bruce De Grazia (Assist. Deputy Under Secretary of Defense)
C9	David Brymer, TNRCC
C10	Silky Labie
C11	Barbara Hill (Waste Management)
C12	Jackie Sample
C13	J.R. Hall (?)
C14	Kodak
C15	Carl Kircher (2nd set of comments)

Mr Slayton will address comments contained in an October 5, 1998 Email from Ms. Cindy Dingman and Mr. Thomas McAninch (Eastman).

Action Items from NELAC IV
Matrix/Medium
References to Matrix/Medium in the chapter:

5.10.1.2 Laboratory Method Manual(s)

b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:
   2) applicable matrix or matrices;

5.10.2.1 Method Validation/Initial Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory initial demonstration of method performance is required.
b) Thereafter, continuing demonstration of method performance (such as laboratory control samples) is required.
c) In all cases, the appropriate forms such as the Certification Statement (Appendix C) or standard performance checklists (see Appendix E) must be completed and retained by the laboratory to be

made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the checklists must be retained by the laboratory.

d) Initial demonstration of method performance must be completed each time there is a significant change in instrument type, personnel, or test method. Comments: If the question is whether or not a change in matrix/medium should require this initial demonstration of performance, my feeling would be that it should. If the question is which term to use then consider this document.

Appendix B

Batch: environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Environmental Detection Limit (EDL): the smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (Radioanalysis Subcommittee)

Laboratory Control Sample (however named, such as laboratory fortified blank or spiked blank): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC).

matrix: The component or substrate which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.
- Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of a industrial process that results in a matrix not previously defined.
- Air Samples: Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix. (Quality Systems)

matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (NELAC).

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Appendix C - INITIAL DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR INITIAL DEMONSTRATION OF CAPABILITY

An initial demonstration of method performance must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method (see 5.10.2.1).

All initial demonstrations, continuing demonstrations and method certification shall be documented through the use of the forms in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed:

a) A quality control sample shall be obtained from an outside source. If not available, the QC check sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The concentrate shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the required method volume to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

Initial Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

matrix:
(examples: laboratory pure water, soil, air, waste solid, leachate, sludge, other)
Method number, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls
   1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

b) Positive Controls
   1) Laboratory Control Sample - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total

suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance. NOTE: the matrix spike (see 2 below) may be used as a control as long as the acceptance criteria are as stringent as the LCS.

2) matrix Spikes (MS) - Shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the test method does not specify the spiking compounds, the laboratory shall spike all reportable components in the Laboratory Control Sample and matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses and shall include permit specified analytes and other client requested components. The laboratory shall ensure, however, that all reported components are used in the spike mixture within a two-year time period, and that no one component or components dominate the spike mixture.

D.1.2 Analytical Variability/Reproducibility

matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Analytical Capability - (Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

D.1.4 Method Detection Limits

Method detection limits (MDL) shall be determined by 40 CFR Part 136, Appendix B unless included in a test method or program.

b) The method detection limit shall be initially determined for the compounds of interest in each test method in a clean matrix appropriate to the test method (such as laboratory pure reagent water or Ottawa sand) or the matrix of interest (see definition of matrix).

d) The MDL shall be verified annually by the preparation and analysis of at least one clean matrix sample spiked at the current reported MDL. If the selected components cannot be detected, the MDL study must be repeated.

e) All procedures used must be documented including the matrix type.

D.4.1 Negative Controls

b) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry the method blank shall be of similar counting geometry that is empty or filled to

similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

D.4.2 Positive Controls

b) matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which do not utilize an internal standard or carrier and for which there is a physical or chemical separation process and where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The matrix spike result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified matrix spike acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~a~~b)19 and 20] will be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11]. The lack of sufficient sample aliquot size to perform a replicate analysis should be noted in the laboratory report.

c) The activity of the laboratory control sample and matrix spike analyte(s) shall be greater than ten times and less than one hundred times the a priori detection limit.

d) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration.

e) Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. isotopic uranium: U-234, -235, and -238) only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified activity level each shall be assessed against the specified acceptance criteria.

f) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g. americium-241), medium (e.g. cesium-137) and high (e.g. cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

D.5 AIR TESTING

Analyses for Air Toxics shall follow the essential quality controls for chemistry outlined in Appendix D.1. For air testing, the blank, laboratory control sample and a desorption efficiency (such as charcoal tubes) shall be used. matrix spikes and duplicate samples shall be used when feasible.

Appendix E - PERFORMANCE BASED MEASUREMENT SYSTEM

RESERVED - The information presented here is the most recent EMMC Workgroup draft, and is provided for information only.

E.1 CHECKLIST OVERVIEW

The Checklists present consensus among EPA's programs on performance "categories" that allow use of the same Checklists across the Agency's various programs/projects. The Checklists may be applied to screening and field techniques as well as traditional laboratory procedures.

Implementation of the Checklists is intended to be program-specific and a category that does not apply within a specific EPA program or project will be indicated by NA (not applicable). Criteria for a specific EPA program or project are to be filled in under the "Performance Criteria" column; e.g., an Office of Water Reference Method may specify 20% RSD or a correlation coefficient of 0.995 for the category that specifies calibration linearity, whereas an Office of Solid Waste project may specify a Measurement Quality Objective of 12% RSD or a correlation coefficient of 0.998 for this category.

For each EA program or project, the checklists are to be completed for each matrix within each medium for which performance is demonstrated.

E.1.1 Header

f) medium: enter the type of environmental sample, e.g., water--NOTE a separate checklist should be prepared for each matrix, e.g., for checklists associated with performance-based methods for SDWA, enter Drinking Water as the matrix type. As the evaluations of a performance-based

method will involve matrix-specific performance measures, a separate checklist would be prepared for each matrix. The medium is the environmental sample type to which the performance-based method applies, whereas the performance category matrix, appearing in the body of the checklists refers to the specific sample type within the medium that was spiked, e.g., for medium hazardous waste, the checklist category matrix may be solvent waste.

E.1.2 EPA PBMS Checklist for Initial Demonstration of Method Performance

The Initial Demonstration of Method Performance involves multiple spikes into a defined sample matrix (e.g., wastewater, paper plant effluent), to demonstrate that the Performance-based Method meets the Program or Project Performance Criteria based on the performance of established Reference Method or based on Measurement Quality Objectives (analytical portion of the Data Quality Objectives). This exercise is patterned after the Initial Demonstration of Capability in C.1 of this appendix.

#10. Interferences.
Enter information on any known or suspected interferences with the performance-based method. Such interferences are difficult to predict in many cases, but may be indicated by unacceptable spike recoveries in environmental matrices, especially when such recovery problems were not noted in testing a clean matrix such as reagent water. The interferences associated with the reference method are to be indicated, as well as, the effect of these interferences on the performance-based method.

#12. Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).
Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria. Where applicable, provide a listing of analytes for which the PE results were "not acceptable".

#20. Method Blank Results.
A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the concentrations of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#21. matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.).
Refers to the specific sample type within the broader medium that was spiked, e.g., for medium: Hazardous Waste an example matrix spiked as part of the initial demonstration of method performance might be "solvent waste".

#22. Spiking System, appropriate to the method and application.
Enter the procedure by which a known amount of analyte/s ("spike") was added to the sample matrix. This may include the solvent that is employed and the technique to be employed (e.g., permeation tube, or volumetric pipet delivery techniques spiked onto a soil sample and allowed to equilibrate 1 day, etc.). Solid matrices and air are often difficult to spike and considerable detailed narrative may be necessary to delineate the procedure. For spikes into aqueous samples generally a water miscible solvent is needed.

#23. Spike concentrations (w/units corresponding to final sample concentration).
Enter the amount of the analyte/s ("spike") that was added to the sample matrix in terms of the final concentration in the sample.

#25. Number of Replicate Spikes.
The initial demonstration of method performance involves the analyses of replicate spikes into a defined sample matrix (category #21). Enter the number of such replicates. For example in the NPDES and SDWA programs, at least 4 replicates should be prepared and analyzed independently.

#28. Detection Limit (w/units; analyte by analyte), if applicable.
A general term for the lowest concentration at which an analyte can be detected and identified. There are various approaches to establishing detection limits which include "Limit of Detection" and 'Method Detection Limit". Enter the approach used (e.g., MDL) and the analytical result with units for each analyte in the matrix (see #21).

#29. Confirmation of Detection Limit. if applicable.
In addition to spikes into the matrix of interest (see #21) it may be beneficial to perform the detection limit measurements in a clean matrix, e.g., laboratory pure water, air, sand, etc. Results of the spikes in the clean matrix are frequently available in the Agency's published methods. Determining MDLs in a clean matrix using the performance-based method will allow a comparison to the MDLs published in the Agency methods.

This performance category is of importance when operating at extremely low concentrations. If the concentrations measured or the decisions to be made, e.g., action levels, are several orders of magnitude above these concentrations, the "quantitation level" should be entered.

Also, the detection limit technique may specify specific procedures to verify that the obtained limit is correct, e.g., the "iterative process" detailed in the 40 CFR Part 136, Appendix B, MDL procedures.

E.1.3 EPA PBMS Checklist for Continuing Demonstration of Capability:

The process by which a laboratory documents that its previously established performance of an analytical procedure continues to meet performance specifications as delineated in this checklist.

#1. Method Blank Result.
A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the levels of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#4. Laboratory Control Sample.
An analytical standard carried through all aspects of the analytical method, e.g., digestions, distillations and determinative steps/instrumentation. It is generally used to assess the performance of all of the measurement system independent of the challenges of the sample matrix.

#6. Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).
Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria.

#11. matrix (reagent water, drinking water, sand, loam, clay, waste solid, ambient air, etc.).
Refers to the specific sample type within the broader "medium" that was spiked, e.g., for medium: Waste an example matrix, spiked as part of the initial demonstration of method performance, might be solvent waste.

#12. matrix Spike Compounds.
Enter the analytes spiked. In preparing a matrix spike, a known amount of analyte is added to an aliquot of a real-world sample matrix. This aliquot is analyzed to help evaluate the effects of the sample matrix on the analytical procedure. matrix spike results are typically used to calculate recovery of analytes as a measure of bias for that matrix.

#13. matrix Spike Concentrations (w/units corresponding to final sample concentration).
Enter the amount of the analyte/s or "spike" that was added to the sample matrix in terms of the final concentration in the sample.

#14. Recovery of matrix Spike (w/units).

#21. matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.).
Refers to the specific sample type within the broader medium that was spiked, e.g., for medium: Hazardous Waste an example matrix spiked as part of the initial demonstration of method performance might be "solvent waste".

The ratio of the standard deviation of a series of at least three measurements to the mean of the measurements. This value is often expressed as a percentage of the mean.

Note: Some programs/projects have utilized matrix spike duplicates (a separate duplicate of the matrix spike) to help verify the matrix spike result and to provide precision data for analytes which are not found in real-world samples, since duplicates of non-detects provides little information concerning the precision of the method. See Item # 19.

EPA PBMS
Checklist for Initial Demonstration of Method Performance

Provide a checklist for each matrix included in the demonstration.
"matrix" include in table

Comments: Media and medium is used consistently in the micro section as "an environment in which something may function or flourish"-Webster's. The implication is that a medium is something used as part of the testing process.

Air sampling, under the matrix definition, uses medium as the filter which is part of the testing process.

Medium is also used in this chapter as a "middle condition:.

Magnetic media is used to describe a mode of records storage.

Matrix does not have a good "non jargon" definition in everyday usage but seems to be accepted in the industry as the "thing" that the analyte of interest resides while in nature.

The EMMC draft of Appendix E seems to be pushing for a tiered approach to matrix through the combination of the terms "medium" and "matrix" which can produce an infinite number of classifications.

Definition found in Utah administrative law: "matrix" means a surrounding substance within which something originates, develops, or is contained, such as: drinking water,

saline/estuarine water, aqueous substance other than drinking water or saline/estuarine water, non-aqueous liquid, biological tissue, solids, soils, chemical waste, and air.

Recommendation:
Media/medium is used a number of different ways in the chapter. Electronic media and microbiology media seem to be accepted usage. It has a separate meaning in these two cases that would become even more ambiguous if media/medium were defined by NELAP or this chapter, as it has been suggested, to have a relationship to matrix.

1 - It may be better to use a different term for "medium" [as used in Appendix E] if the distinction needs to be made.

or

2 - The definition for matrix could be expanded to include the categories that EMMC has envisioned in Appendix E an eliminated any reference to medium as used in Appendix E.

HOMEWORK

The next homework is from the Action items from NELAC IV.

Committee to decide if there is a need to exempt preservation checks in certain circumstances and to identify specific standards if required (Section 5.11.3) SheilaM

First of all, the first and second sentences in 5.11.3 contradict each other. The first sentence states that all samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within ***+/- 2 degrees of the required temperature or the method specified range.*** For example if the required temperature is 4 degrees, the acceptable range would be 2 - 6 degrees. However, in the second sentence it states, that for samples with a specified temperature of 4 degrees (which in the above sample could be a required temperature) a temperature range of 0.1 - 6 degrees in now acceptable!!! There is further confusion by allowing additional leeway +/- 2 degrees to an already established temperature range in the first sentence. For example, if a method specifies an acceptable range as .1 - 6 degrees, in addition we will allow +/- 2 degrees on top of that.

I believe we have already exempted preservation checks (requirements) for thermal preservation for samples collected shortly before the arrival to the laboratory, by requiring evidence that the chilling process has begun. This of course would be indicated on the COC form. **I think we need to stay away from a time requirement for getting samples from the field to the lab unless someone has data to demonstrate how long it takes samples in an ice chest to reach a specified temperature.**

For checking chemical preservation, our standards state that the laboratory shall implement procedures **"prior to or during sample preparation or analysis,"** which as states in 5.11.2 would be in the laboratory's acceptance policy. I think additional language could be added to state that confirmatroy preservation checks are not required when: **appropriate method of preservation has been documented as specified in a test procedure or SOP.**

HOMEWORK

Homework #C9

Issue from David Brymer, TNRCC, 512-239-1725

Issue regards 5.9.4.4.2.a (Continuing Calibration Verification) which currently reads:

> These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be the standards used in the original calibration curve or standards from another source. The frequency shall be increased if the instrument consistently drifts outside acceptance criteria before the next calibration.

Comments:

> Mr. Brymer's concern seems to be in the area of continuous monitoring systems and how the language above would impact the frequency with which these systems are subject to a CCV. If a continuous monitoring system were to acquire a sample/ data point every 5 minutes then a CCV would be required on the order of every 2 hours. This when such systems are currently allowed to run CCVs once per day or even once per week.

Suggestion:

> In addressing 5.9.4 in Annapolis we should address whether the CCV frequency should be left up to the respective method or if we should specify some minimal frequency. And in the case of the latter whether we should give the continuous monitoring community some specific leeway on this issue.